



National Institute of Standards & Technology

Certificate of Analysis

Standard Reference Material[®] 3064

Endothall in Water

This Standard Reference Material (SRM) is a solution of endothall (Chemical Abstracts Registry Number 145-73-3) in water. This SRM is intended primarily for use in the calibration of chromatographic instrumentation used for the determination of endothall. SRM 3064 can also be used to fortify aqueous samples with known amounts of endothall. A unit of SRM 3064 consists of five 2-mL ampoules, each containing approximately 1.2 mL of solution.

Certified Concentration of Endothall: The certified concentration given below is based on results obtained from the gravimetric preparation of this solution and from the analytical results determined by using gas chromatography. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Endothall	40.0 ± 1.1 mg/kg
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The results are expressed as the certified value \pm the expanded uncertainty. The certified value is the unweighted average of the concentrations determined by gravimetric and chromatographic measurements. The expanded uncertainty, at the 95 % level of confidence, is calculated as $U = ku_c$, where u_c is a combined standard uncertainty calculated according to the ISO Guide [1] and $k = 2$ is the coverage factor. The value of u_c includes both a correction for estimated purity and an allowance for differences between the concentration determined by gravimetric preparation and chromatographic measurements.

Expiration of Certification: The certification of this SRM lot is valid until **31 March 2013**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is nullified if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Return of the attached registration card will facilitate notification.

Coordination of the technical measurements leading to the certification was under the direction of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Analytical measurements of the SRM were performed by M.M. Schantz and C.R. Mack of the NIST Analytical Chemistry Division.

Support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the Measurement Services Division.

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Certificate Issue Date: 14 May 2003

John Rumble, Jr., Chief
Measurement Services Division

Preparation of the SRM were performed by M.P. Cronise of the NIST Measurement Services Division and by M.M. Schantz and C.R. Mack of the NIST Analytical Chemistry Division.

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Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh of the NIST Statistical Engineering Division.

NOTICE AND WARNING TO USERS

Handling: This material should be handled with care. Use proper disposal methods.

Storage: Sealed ampoules, as received, should be stored in the dark at temperatures lower than 30 °C.

INSTRUCTIONS FOR USE

Sample aliquots for analysis should be withdrawn at 20 °C to 25 °C **immediately** after opening the ampoules and should be processed without delay for the certified value to be valid within the stated uncertainty.

PREPARATION AND ANALYSIS¹

The endothall monhydrate used in the preparation of this SRM was obtained from a commercial source. The solution was prepared at NIST by weighing and mixing the endothall monhydrate into the water. The weighed endothall monhydrate was added to the water and mixed until completely dissolved and homogenized. The total mass of this solution was measured. The gravimetric concentration was adjusted for the consensus purity estimation of the endothall, which was determined using capillary gas chromatography with flame ionization detection. This bulk solution was then chilled and 1.2-mL aliquots were dispensed into 2-mL amber glass ampoules, which were then flame sealed.

Aliquots from nine ampoules, selected randomly, were analyzed in duplicate by using capillary gas chromatography with flame ionization detection employing an immobilized non-polar (100 % dimethylpolysiloxane) stationary phase column. An internal standard solution containing dicamba was added to each sample for quantification purposes followed by the addition of acidified acetone as detailed by Carlson et al. [2]. Calibration solutions consisting of weighed amounts of endothall and internal standard in water plus acidified acetone were chromatographically analyzed to determine the response factor for endothall relative to dicamba.

¹Certain commercial equipment, instruments, or materials are identified in this certificate in order to specify adequately the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

- [1] *Guide to the Expression of Uncertainty in Measurement*; ISBN 92-67-10188-9, 1st Ed., ISO, Geneva, Switzerland, (1993); see also Taylor, B.N.; Kuyatt, C.E.; *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*; NIST Technical Note 1297, U.S. Government Printing Office, Washington, DC (1994); available at <http://physics.nist.gov/Pubs/>.
- [2] Carlson, R.; Whitaker, R.; Landskov, A.; *Endothall in Analytical Methods for Pesticides and Plant Growth Regulators: New and Updated Methods*; G. Zweig, Ed., Chapter 31, pp. 327-340. Academic Press. New York, NY (1978).

Users of this SRM should ensure that the certificate in their possession is current. This can be accomplished by contacting the SRM Program at: telephone (301) 975-6776; fax (301) 926-4751; e-mail srminfo@nist.gov; or via the Internet <http://www.nist.gov/srm>.